Effect of Lamination Conditions on the Gas Permeability and Adhesion Strength of Green Ceramic Tapes

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Abstract

The gas permeability and adhesion strength of laminated green ceramic tapes were determined for samples comprised of barium titanate as the dielectric, and poly(vinyl butyral) and dioctyl phthalate as the main components of the binder mixture. The green tapes were laminated for times of 2-10 min, pressures of 1.8-7 MPa, and temperatures of 35-85°C. The adhesion strength, which was measured by a peel test, increased with increasing lamination time, temperature, and pressure. The permeability, which was determined from gas flux measurements, decreased with increasing lamination time, temperature, and pressure. The dependence of the permeability and adhesion strength on lamination time, temperature, and pressure is qualitatively consistent with a mechanistic description of the lamination process as one of binder flow in porous media

INTRODUCTION

The lamination of green tapes is one of the steps in the fabrication of multilayered ceramic components. This processing step influences a number of properties of the green and sintered ceramic body, such as the density, interlayer adhesion, and microstructure, including evidence of the lamination interface. Several studies have examined these properties after the lamination step, primarily in relation to the binder type and binder content [1-7].

In addition to binder type and amount influencing the properties of the multilayered body, the specific conditions of the lamination process, namely the lamination time, temperature, and applied load, are also expected to influence material properties. For example, the laminate bond strength has been shown to increases with the logarithm of the product of the lamination time, temperature, and pressure [1]. Although it may be expected that other properties vary with the conditions of lamination, less work has appeared that systematically examines the relationship between the conditions of lamination and the properties of the green ceramic body. It is also probable that a set of lamination parameters which leads to an improvement in one property will lead to deleterious consequences in another property.

Two properties that may exhibit such a trade-off in behavior depending on the conditions of lamination are the gas-phase permeability [8-11] and the interlayer adhesion strength [1,6],

and a trade-off between these two properties may have a pronounced effect on the subsequent processing of the laminated ceramic body, especially in the thermal removal of binder. High permeability after lamination is desirable to minimize the buildup of pressure during binder removal, which can thereby lead to higher product yield and to shorter binder removal heating cycles. From the aspect of interlayer adhesion, a high value after lamination is desirable for avoiding delamination during binder removal and for promoting a high degree of bonding between layers during sintering.

In earlier work [10,11], we have shown that the gas-phase permeability of open pore laminated tapes is substantially less than that of unlaminated tapes at one set of lamination conditions. A detailed examination of the influence of the lamination time, temperature, and pressure on the permeability, however, was not conducted. Although the effect of the lamination conditions on the interlayer adhesion strength has been examined [1], parallel measurements on the permeability for the same ceramic and binder system were not been reported. It is also of interest to examine if the reported dependence of the interlayer strength on the logarithm of the lamination time, temperature and pressure is also exhibited in another ceramic powder and binder system for both the adhesion strength and the permeability.

In light of the above considerations, we systematically examine in this work the trade-off

between permeability and adhesion strength as a function of the lamination conditions of time, temperature, and pressure. In addition, we show the effect that the orientation of tapes during lamination has on the permeability and adhesion strength of laminated tapes. The observed dependence of these two properties on the lamination conditions is then rationalized qualitatively in terms of a flow model for describing the process of lamination.

EXPERIMENTAL

Green ceramic tapes were prepared from barium titanate powder (Tamtron X7R 412H, Ferro Electronic Materials, Niagara Falls, NY), which has a mean particle diameter of 1.2 μm and specific surface area of 3.3 m^2/g. The slurry preparation consisted of mixing 55.4 weight% barium titanate powder with 44.6 weight% of a binder solution (B73305 Ferro Corp., San Marcos, CA), which is 22% by weight poly(vinyl butyral) (PVB) and dioctyl phthalate (DOP) in a 6:4 weight ratio in a solvent mixture. The slurry was ball milled, filtered, and then tape cast on a Mylar film. The tapes after drying had a thickness of approximately 165 μm. The total organic content of the substrates was 10.9% by weight, as determined by weight loss.

The tape samples were laminated using a 25 ton press (Model 2518, Carver, Inc., Wabash, IN) with heated platens over a range of times (2-10 min), temperatures (20-85°C), and nominal

pressures (1.8-7MPa). Two tapes were placed on the lower platen, which was already at the desired temperature, and then the load was applied and held for a fixed amount of time. The load was converted to a pressure by using the nominal area of the faces of the tapes.

The gas permeability measurements [10,11] (see Fig. 1) were performed by placing two laminated green tapes of disk geometry into a sample holder with an open area of 1.56 cm in diameter. The upstream pressure of nitrogen, P_1=0.5 MPa, was set by a pressure regulator, and the downstream pressure was set at P_2=0.1 MPa. The volumetric flow rate, V_f, which was measured by a flow meter, was converted to a molar flow rate, n, via

$$n = \frac{P_f V_f}{RT_f} \qquad 1$$

where R is the gas constant, T is the temperature, and the subscript f denotes the conditions at the flow meter. The measured molar flux, N_m, through the area, A, of the substrate is then given by

$$N_m = n / A = u_o \rho \qquad 2$$

where u_o is the superficial velocity and $\rho = P / RT$ is the molar density. For gas flux governed by contributions from Knudsen, slip, and Poiseuille flow, it has been shown that the gas permeability, K, can be represented from the measured flux data as [12,13]

$$K = -RTN_m \frac{L}{P_2 - P_1} \qquad 3$$

where L is the sample thickness.

To prepare samples for peel tests [14,15], tapes were laminated as indicated in Fig. 2. Green tapes of 2 cm width × 10 cm length were partially separated with a layer of Mylar of 0.08 mm thickness and then were laminated in the press. The two parts of the T-shaped sample not laminated were then placed in grips and subjected to a load with a tensile tester (TA TX Plus Texture Analyzer, Stable Micro Systems Ltd., Surrey, UK). During testing, the top crosshead moved and the load was measured as a function of displacement for a constant crosshead speed of 0.4 cm/s. For the case where the extension due to strain is negligible, the adhesion strength, σ, is given by

$$\sigma = \frac{F_1}{b} \qquad 4$$

where F_1 is the applied force and b is width of sample.

RESULTS

Microstructure

The microstructures of the top and bottom as-cast surfaces of the green tapes are shown in Fig. 3. Pores or channels of irregular shape appear on both surfaces, and some of these pores exceed in size the diameter of the ceramic particles. Although no large difference is observed between the two microstructures, the bottom surface is distinctly different as evidenced by smeared binder on the surface of the tape that was in contact with the Mylar carrier film. This

type of difference was also observed in another study [5] for alumina tapes prepared from a solvent mixture.

For microstructural examination of the laminate interface, samples were prepared by freezing green laminated substrates in liquid nitrogen and then fracturing them. Figure 4 shows the microstructures and interfaces of samples laminated at aggressive conditions (7 MPa at 85°C for 10 min), intermediate conditions (3.5 MPa at 65°C for 5 min), and at mild conditions (1.8 MPa at 50°C for 2 min). For all three sets of lamination conditions, the interface between the layers is almost always evident, although it appears most pronounced in the sample laminated at the mildest set of conditions. For this latter set of conditions, the two tape surfaces also appear at higher magnification not to be uniformly in contact at the interface, i.e., a gap of ~10 μm between tapes is sometimes visible.

To examine the microstructures of cross-sections of laminated tapes that had been sintered, samples were prepared by slicing and then polishing. The microstructures of sintered samples laminated at the most aggressive conditions (7 MPa at 85°C for 10 min), intermediate conditions (3.5 MPa at 65°C for 5 min) and mild conditions (1.8 MPa at 50°C for 2 min) are shown in Fig. 5. The presence of the lamination line is often evident for the tapes laminated at the least aggressive conditions, sporadically evident at the intermediate lamination conditions,

and not at all visible for the tapes laminated at the most aggressive conditions.

The presence or absence of the lamination interface has been examined in other studies as well. In one study, the interface was visible in green tapes laminated at aggressive conditions from a styrene-acrylic latex binder [6], whereas the interface was not visible in the green state when an emulsion-based binder was used [7]. In light of these mixed results, it can be concluded that examination of green tapes is not always a foolproof method for assessing the degree of bonding at the interface.

Examination of sintered samples, as compared to green samples, however, is more informative as to the degree of bonding at the interface. This work, for example, showed clearly that the interface was not evident for samples laminated at aggressive conditions, and another study [7] reported similar behavior as well. For other ceramic, binder, and lamination conditions, however, studies have shown that remnants of the interface may persist through sintering [3,6], and thus for these systems, further optimization of the lamination process may be needed to achieve more intimate bonding at the interface.

Permeability

The gas permeability is shown in Fig. 6 for two tapes laminated at 7 MPa at 85°C for 10 minutes. Individual values of the permeability of 5 specimens are shown, along with the average,

for experiments conducted with different tape orientations during lamination of top face to top face (t-t), top face to bottom face (t-b), and bottom face to bottom face (b-b). Tapes laminated in a t-b fashion exhibit the lowest permeability and also the most uniformity as compared to tapes laminated in t-t and b-b fashions. For the remaining permeability measurements, all samples were laminated in a t-b fashion.

Figure 7a illustrates the permeability of tapes laminated at 7 MPa as a function of lamination temperature for different lamination times. At low lamination temperatures, the permeability does not depend strongly on either the lamination temperature or time. With increasing lamination temperature above 35°C, the permeability begins to decrease more strongly. Lamination times of 2-5 min do not lead to pronounced differences in the permeability over the temperature range examined here. This lack of sensitivity to shorter times may arise because an initial period of time is needed for the tapes to become uniformly in contact at the interface (see Fig. 4). For tapes laminated for 10 minutes at 7 MPa at 85°C, a reduction in the permeability of nearly three orders of magnitude is evident as compared to tapes laminated at the least aggressive conditions.

Figure 7b shows how the permeability varies with lamination temperature for different lamination pressures for lamination times of 10 min. Once again, the permeability decreases strongly at higher temperatures, and this decrease is most pronounced at a lamination pressure of

7 MPa.

The trends in Fig. 7 thus indicate that the permeability depends sensitively on the conditions of lamination. We note that for the poly(vinyl butyral) binder system examined here, the glass transition temperature is below 10°C [16,17], and thus in spite of being substantially above this temperature, the tape samples, as evidenced by the permeability, only undergo a significant degree of lamination for more aggressive lamination times, temperatures, and pressures. The behavior in Figs. 7a and 7b thus may indicate that only a relatively narrow region of parameter space leads to a sufficient degree of lamination. For even more aggressive lamination conditions, the sample may begin to flow excessively in the lateral directions.

Adhesion Strength

Adhesion strength versus displacement curves are shown in Fig. 8 for two tapes laminated at 7 MPa at 85°C for 10 minutes. For each curve, which is the average of 3-5 samples, the tapes were laminated with different orientations of t-t, t-b, and b-b. Initially, for all three lamination orientations, the adhesion strength rises in a linear manner, reaches a local maximum, and then experiences a relatively constant value versus displacement. Near the end of each test, local or global maxima or minima are also observed. The nominal adhesion strength can be taken as the initial maximum achieved, although the plateau regions are also

indicative of the same ordering in adhesion in that the highest adhesion strength corresponds to the samples laminated in a b-b fashion, and the lowest adhesion strength arises from samples laminated in a t-t fashion. Thus, in contrast to the permeability results in Fig. 6, the adhesion strength for samples laminated in the t-b fashion is intermediate between those of samples laminated in a t-t and b-b fashion. Although not shown here, a value at failure of ~400 N/m was obtained for tape samples tested in pure tension, and in addition, the slope of the curve of F_1/b versus displacement was at least 5 times larger than the initial slopes shown in Fig. 8. Thus, the individual tapes are much stronger and yield less than the lamination bond.

The average adhesion strength versus displacement curves for 3-5 samples laminated at different pressures in a t-b fashion at 85°C for 10 min are shown in Fig. 9a. With increasing lamination pressure, the adhesion strength increases by approximately a factor of three over the range of conditions examined here. Figure 9b shows the effect of lamination temperature on the adhesion strength versus displacement for samples laminated at 7 MPa for 10 minutes. With increasing lamination temperature, the adhesion strength increases by a factor of three over the range of conditions examined here. The effect of lamination time is illustrated in Fig. 9c for samples laminated at 7 MPa at 85°C, where with increasing lamination time, the adhesion strength increases. The results in Fig. 9 thus indicate that large differences arise in the adhesion behavior depending on the lamination conditions, and that the initial maximum in the adhesion

strength is a good indicator of the bonding between layers.

DISCUSSION

Figure 10 summarizes the trade-off in behavior for the adhesion strength and permeability versus one parameter of lamination when the other two parameters are held constant. More aggressive lamination conditions of time, temperature, and pressure lead to better adhesion between layers, which may lead to improved yield during binder removal. These same lamination conditions, however, lead to less permeability in the green body, with concomitant slower heating cycles necessary to avoid pressure buildup and failure.

To examine the dependence of the adhesion strength and permeability in terms of more than one lamination variable, we proceed in the following fashion. Earlier work has shown that the interlayer strength between laminated tapes depends logarithmically on the conditions of lamination [1], and thus we use models of the form

$$\sigma = A + B\ln(CPTt)\,; \qquad K = A + B\ln(CPTt) \tag{5}$$

For comparison, we use two other models given by

$$\ln\sigma = At + BT + CP + D\,; \qquad \ln K = At + BT + CP + D \tag{6}$$

$$\sigma = A\left[\frac{Pt}{\exp(B/T)}\right]^{C}; \qquad K = A\left[\frac{\exp(B/T)}{Pt}\right]^{C} \tag{7}$$

The constants A-D are then determined by nonlinear regression analysis for both the adhesion

strength and the permeability, and Table 1 summarizes the constants along with the average error and the regression coefficient, R^2, for each model. For the adhesion strength, the best model is Eq. 7, which predicts the adhesion strength to within an average error of 9%. For the permeability, Eq. 6 predicts to within 50% of the measured values over three orders of magnitude. Equation 5 is the poorest of the models for describing how the adhesion strength and permeability vary with lamination conditions for the samples evaluated in this work. Equations 6 and 7 are thus both reasonably accurate over a broad range of parameter space, and are useful to describe the dependence of the adhesion strength and permeability on the lamination conditions.

Figure 11a shows the dependence of the adhesion strength determined from Eq. 7 versus lamination time and temperature for a fixed lamination pressure of 7 MPa. The contours of this graph show that if a minimum adhesion strength of say 30 N/m is required, then only a relatively narrow range of lamination conditions of t>8 min and T≈80°C are available to satisfy this condition for a fixed P=7 MPa. Outside of this narrow operating window, for example, say at t=6 min, it would be necessary to raise the temperature substantially above 80°C to achieve 30 N/m; at such elevated temperatures, however, thermal decomposition of the binder would then begin to occur [18]. The narrowness of this processing window arises because, as seen in Eq. 7, the adhesion strength for a fixed pressure depends on the ratio of two operating variables, namely

the lamination time and an exponential dependence on temperature. Figure 11b shows similar behavior for the adhesion strength as a function of lamination time and pressure for a fixed lamination time of 10 min. Once again, steep contours are seen for the adhesion strength, which again depends on the ratio of the dependences on lamination pressure and temperature.

Figure 12a shows the permeability determined from Eq. 6 as a function of the lamination time and temperature for a fixed lamination pressure of 7 MPa. If a fixed permeability of say greater than 1×10^{-7} m^2/s is desired, then, once again, because of the functional dependence given by Eq. 6, only a narrow region of parameter space is available to achieve this value for P=7 MPa. In a similar fashion, Fig. 12b shows similarly steep contours for the permeability versus the lamination pressure and temperature for a fixed lamination time of 10 min.

The results in Figs. 10-12 indicate that in practice, it may not be possible to simultaneously satisfy specifications on both the properties of permeability and adhesion strength. This arises in part because of the difficulty in identifying the narrow region of operating space to achieve one property and because of the inverse dependences of the two properties on the conditions of lamination. It may be for this reason that in practice, measures pertaining only to the degree of lamination are typically used to identify a successful lamination process, or alternatively, changes to the binder system or binder amount are needed.

In light of the dependences of the adhesion strength and permeability on the lamination

time, temperature, and pressure, a plausible mechanistic model for the lamination process is one of pressure driven flow of binder in a porous medium. Figure 13 schematically represents this viewpoint of two tapes prior to lamination and denotes the relevant length scales of an initial gap length, L_g, of the tapes in loose physical contact, a penetration depth, L', of binder, and a pore diameter, D. The binder is assumed to flow with a viscosity, μ, which varies with temperature according to an activated process as $\mu = k \exp(E/RT)$. Initially, as the load is applied to the tapes, the gap length, L_g, goes to zero, and then as the tapes become in intimate contact, a pressure drop, $\Delta P / L'$, leads to binder flow in the pore space surrounding each particle, including flow across the interface; slight particle rearrangement may also occur. Dimensional analysis [19] of the binder flow process in terms of the quantities L', t, μ, D, and $\Delta P/L'$ indicates that the penetration depth will be a function of the dimensionless group $[D(\Delta P/L')t/\mu]$, which contains the same ratio in terms of time, pressure, and temperature, via the viscosity, as in Eq. 7. If the penetration depth of binder flow is then taken as proportional to the adhesion strength, and inversely proportional to the permeability, then the regression analysis results in Table 1 lead to

$$L' \propto \sigma = 57.3 \left[\frac{Pt}{\exp(3160/T)} \right]^{1.0}; \qquad 8$$

$$\frac{1}{L'} \propto K = 2.97 \times 10^{-9} \left[\frac{\exp(4350/T)}{Pt} \right]^{0.65} \qquad 9$$

For comparison, the Washburn equation for pressure driven flow in capillaries leads to the

penetration depth as

$$L' = \left[-\frac{D^2 \Delta P t}{16 \mu} \right]^{1/2} \qquad 10$$

which has the same dependence on pressure, time, and temperature as Eq. 8. The exponent in Eq. 9 for the permeability agrees more closely with the behavior described by the Washburn equation as compared to the adhesion strength.

Although the mechanism presented above is undoubtedly simplified, it does provide a plausible description of the lamination process in terms of the adjustable parameters of lamination time, temperature, and pressure. Modeling of the lamination step as a flow process is also consistent with the observation that as the conditions of lamination become too extreme, the lateral dimensions of the multilayer sample become larger and the thickness becomes smaller. Although other mechanisms such as diffusive entanglement and chemical reaction between the polymer molecules may also occur, it is likely that the flow aspect of the lamination process is what ultimately leads to intimate bonding at the interface and to the disappearance of interface during sintering.

CONCLUSIONS

The gas permeability and adhesion strength of laminated green tapes have been determined as a function of the lamination conditions. With increasing lamination time, temperature, and pressure, the permeability decreases whereas the adhesion strength increases, thus indicating a trade-off between these two properties and the conditions of lamination. A mechanism based on binder flow in porous media can be used to rationalize the observed dependences of adhesion strength and permeability on lamination time, temperature, and pressure.

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Table 1 Model constants for Eqs. 5-7, average error, and regression coefficients for describing the adhesion strength (N/m) and permeability (m^2/s) versus lamination conditions in units of t (s), T (K), and P (MPa).

Model Equation	A	B	C	D	Average Error (%)	R^2
σ (N/m)						
5	-2.55	10.91	7.73×10^{-6}	-	28	0.53
6	3.25×10^{-3}	0.0356	0.293	-13.03	13	0.93
7	57.3	3160	1.0	-	9	0.97
κ (m^2/s)						
5	1.05×10^{-6}	-6.73×10^{-8}	2.36	-	423	0.28
6	-3.23×10^{-3}	-0.0625	-0.65	9.14	50	0.88
7	2.97×10^{-9}	4350	0.65	-	402	0.43

Figure Captions

1. Schematic of the apparatus used to determine the permeability of green ceramic tapes.
2. a) Schematic of the procedure used to laminate the green ceramic tapes. b) Schematic of how the peel test was conducted. In both panels, the arrows indicate the direction of the applied force.
3. Scanning electron micrographs of as-cast green tapes showing the a) top surface and b) bottom surface.
4. Scanning electron micrographs of the cross sections of green tapes laminated at a) aggressive conditions (7 MPa, 85 °C, 10 min); b) intermediate conditions (3.5 MPa, 65 °C, 5 min); and c) mild conditions (1.8 MPa, 50 °C, 2 min).
5. Scanning electron micrographs of the cross sections of sintered tapes which were laminated at a) aggressive conditions (7 MPa, 85 °C, 10 min); b) intermediate conditions (3.5 MPa, 65 °C, 5 min); and c) mild conditions (1.8 MPa, 50 °C, 2 min).
6. Permeability values and average permeability versus tape orientation for tapes laminated at 7 MPa at 85 °C for 10 min. Tape orientations of top to top (t-t), bottom to bottom (b-b), and top to bottom (t-b) were examined.
7. a) Permeability versus temperature of tapes laminated a) at 7 MPa for different times, and b) for 10 min at different pressures.

8. Adhesion strength versus displacement for tapes laminated at 7 MPa at 85 °C for 10 min in different orientations. The curves are the averages of 3-5 samples. Tape orientations of top to top (t-t), bottom to bottom (b-b), and top to bottom (t-b) were examined.
9. Average adhesion strength versus displacement for 3-5 tapes laminated at a) different pressures at 85°C for 10 min, b) at different temperatures at 7 MPa for 10 min, and c) at different times at 7 MPa at 85 °C.
10. Average adhesion strength and permeability versus displacement for tapes laminated at a) different pressures at 85°C for 10 min, b) at different temperatures at 7 MPa for 10 min, and c) at different times at 7 MPa at 85 °C.
11. a) Adhesion strength of green tapes as function of time and temperature for tapes laminated at P=7 MPa. b) Adhesion strength of green tapes as function of temperature and pressure for tapes laminated for t=10 min.
12. a) Permeability of green tapes as function of time and temperature for tapes laminated at P=7 MPa. b) Permeability of green tapes as function of temperature and pressure for tapes laminated for t=10 min.
13. Schematic of the lamination process indicating solid particles, binder, pores, and the length scales of pore diameter, D, binder penetration depth, L', and gap length, L_g.